JF 2-2797/2

# JAPANESE PATENT OFFICE PATENT JOURNAL (A)

# KOKAI PATENT APPLICATION NO. HEI 2[1990]-279729

Int. Cl.5:

C 08 G 81/02

77/442

Sequence Nos. for Office Use:

7142-4J 6609-4J

Filing No.:

Hei 1[1989]-101248

Filing Date:

April 20, 1989

Publication Date:

November 15, 1990

No. of Claims:

13 (Total of 11 pages)

Examination Request:

Not filed

# METHOD FOR MANUFACTURE OF MODIFIED POLYSTYRENE RESIN AND IMPACT-RESISTANT POLYSTYRENE RESIN COMPOSITIONS

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#### [Attached amendments have been incorporated into text of translation]

#### Claim

- 1. A method for manufacture of modified polystyrene resin, characterized in that a polystyrene elastomer modified with maleic anhydride is reacted with polysiloxane having a reactive group.
- 2. A method for manufacture of modified polystyrene resin as in Claim 1, wherein the polystyrene elastomer modified with maleic anhydride is obtained by selectively hydrogenating the polybutadiene segment of a styrene-butadiene block copolymer (where the number of polystyrene segments and polybutadiene segments is at least one, respectively) and grafting with maleic anhydride.
- 3. A method for manufacture of modified polystyrene resin as in Claim 2, wherein the styrene-butadiene block copolymer contains 15-60 wt% of polystyrene segment.
- 4. A method for manufacture of modified polystyrene as in Claim 1, 2, or 3, wherein the content of maleic anhydride is 20 wt% or less.
- 5. A method for manufacture of modified polystyrene as in Claims 1-4, wherein the polysiloxane having a reactive group is amino group-terminated (at one end) polysiloxane with a weight-average molecular weight of 1000-100,000.
- 6. A method for manufacture of modified polystyrene as in Claims 1-4, wherein the polysiloxane having a reactive group is amino group-terminated (at both ends) polysiloxane with a weight-average molecular weight of 1000-100,000.
- 7. A method for manufacture of an impact-resistant polystyrene composition, characterized in that the modified polystyrene resin as in any of Claims 1-6 is mixed with polystyrene or styrene copolymer.
- 8. A method for manufacture of an impact-resistant polystyrene composition, characterized in that a polystyrene elastomer modified with maleic anhydride is reacted with polysiloxane having a reactive group in the presence of polystyrene or styrene copolymer.
- 9. A method for manufacture of an impact-resistant polystyrene composition as in Claim 7 or 8, wherein the polystyrene elastomer modified with maleic anhydride is obtained by selectively hydrogenating the polybutadiene segment of a styrene-butadiene block copolymer (where the number of polystyrene segments and polybutadiene segments is at least one, respectively) and grafting with maleic anhydride.
- 10. A method for manufacture of an impact-resistant polystyrene resin composition as in Claim 9, wherein the styrene-butadiene block copolymer contains 15-60 wt% of polystyrene segment.

- 11. A method for manufacture of an impact-resistant polystyrene resin composition as in Claim 7, 8, 9, or 10, wherein the content of maleic anhydride is 20 wt% or less.
- 12. A method for manufacture of an impact-resistant polystyrene resin composition as in Claims 7-11, wherein the polysiloxane having a reactive group is amino group-terminated (at one end) polysiloxane with a weight-average molecular weight of 1000-100,000.
- 13. A method for manufacture of an impact-resistant polystyrene resin composition as in Claims 7-12, wherein the polysiloxane having a reactive group is amino group-terminated (at both ends) polysiloxane with a weight-average molecular weight of 1000-100,000.

#### Detailed explanation of invention

Industrial application field

The present invention relates to polystyrene resins modified by grafting with polysiloxane and its composition.

#### Prior art

Polystyrene resin is hard, has excellent high frequency electric insulation property, is strong against acid and alkali, and is easily injection molded since it starts to soften around 90°C and becomes a viscous liquid around 130°C. On the other hand, besides the drawback of brittleness, it has problems in mold release, wear resistance, and lubricating property, since the sliding characteristic such as lubricating property at the molding surface is insufficient.

Among the aforementioned drawbacks of conventional polystyrene resin, the brittleness has been improved by development of high-impact polystyrene, ABS resin, ASA resin, etc., and the sliding characteristic has been improved by the following methods.

- 1) Spraying of silicone oil such as polydimethylsiloxane on molds during molding,
- 2) Blending of silicone oil such as polydimethylsiloxane,
- 3) Copolymerization of styrene with olefin compounds having polydimethylsiloxane chain,
- 4) Blending a copolymer of styrene and olefin compounds having a polydimethylsiloxane chain with a styrene resin having no other polysiloxane chain in the constituent unit.
  - 5) Block copolymerization of a polystyrene unit and polysiloxane unit,
- 6) Blending of a polystyrene unit-polysiloxane unit copolymer with a styrene resin having no other polysiloxane chain in the constituent unit,
  - 7) Graft copolymerization of a styrene monomer to a polysiloxane chain,
- 8) Blending of a copolymer obtained by grafting a styrene monomer to a polysiloxane chain and a styrene resin having no other polysiloxane chain in the constituent unit.

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Problems to be solved by invention

In the prior art, the method for spraying silicone oil on molds has the drawbacks that oil sprayed as a mold release agent remains on the surface of products, spraying of a mold release agent on molds is troublesome, and it is inefficient, besides not being useful for improvement of the sliding characteristic.

Polystyrene resins modified by blending with silicone oil have many problems such as the silicone oil is easily separated during mixing or kneading, bleeding out easily occurs, it does not homogeneously mix, and it causes degradation of physical properties, especially reduction of strength, since it has poor miscibility with resins.

Copolymers of styrene with olefin compounds having a polydimethylsiloxane chain can be synthesized by radical and anion copolymerization as well as the method described in a paper by Yuhsuke, Kawakami, R.A.N. Murthy, and Yuya Yamashita (Die Makromolekulare Chemie, Vol. 185, p. 9 (1984)), but in the present method, its polymerization degree and yield are insufficient, and a large amount of homopolystyrene is produced as a byproduct and its removal is difficult, thus it is difficult to commercially produce the copolymers by those methods. Thus, it is also difficult to blend this kind of copolymer with other resins.

Block copolymers possessing polystyrene segments and polysiloxane segments are obtained by methods utilizing basic monofunctional initiators such as butyllithium described in a paper by J.C. Saan, D.J. Gardon, and S. Lindsay (Macromolecules, Vol. 3, p. 1 (1970)), a paper by A. Marsiat and Y. Gallot (Die Makromolekulare Chemie, Vol. 176, p. 1641 (1975)), a paper by J.G. Zillox, J.E.L. Roovers and S. Bywater (Macromolecules, Vol. 8, p. 573 (1975)), etc., or bifunctional initiators such as sodium naphthalene and lithium naphthalene described in a paper by D.S. Brown, K.U. Fulcher and R.E. Wetton (Journal of Polymer Science Polymer Letter Edition, Vol. 8, p. 659 (1970)), a paper by J.W. Dean (Journal of Polymer Science Polymer Letter Edition, Vol. 8, p. 677 (1970)), a paper by D.J. Legrant (Journal of Polymer Science B, Vol. 8, p. 195 (1970)), a paper by H.G. Kim (Macromolecules, Vol. 5, p. 594 (1972)), a paper by C. Price, A.G. Warson, and M.T. Chov (Polymer, Vol. 13, p. 333 (1972)), a paper by F.R. Jones (European Polymer Journal, Vol. 10, p. 249 (1974)), etc., and a method of anion polymerizing styrene using lithium biphenyl as an initiator described in a paper by P. Bajaj, S.K. Varshney, and A. Misra (Journal of Polymer Science Polymer Chemistry Edition, Vol. 18, p. 295 (1980)) and copolymerizing with hexamethylcyclotrisiloxane or octamethylcyclotetrasiloxane, etc., but they have drawbacks of long reaction time besides requiring the necessary reaction apparatus, and furthermore, since it is difficult to maintain proper reaction conditions, control of the molecular weight of each polystyrene segment and polysiloxane segment and also control of the molecular weight of the final products are difficult. In addition, the polymer species which can be

synthesized are limited, and it is difficult to obtain products having the desired compositions, and thus it is also difficult to blend this kind of copolymer with other resins. This kind of block copolymer can also be obtained by condensation reaction of polystyrene, both ends of which are modified by silanol, with polysiloxane possessing chlorinated functional groups at both ends as described in a paper by A. Skoulios (Macromolecules, Vol. 4, p. 268 (1971)), but, in this method, besides a different number of segments being produced, control of the molecular weight of each polystyrene segment and polysiloxane segment and control of the molecular weight of the final products are difficult, the polymer species which can be synthesized are limited, and it is difficult to obtain products having the desired compositions, thus it is also difficult to blend this kind of copolymer with other resins.

Graft copolymers of styrene to polysiloxane chains possess the possibility of becoming materials having good mold release property, wear resistance, and lubricating property, but in the conventional method for synthesis of this graft copolymer, they are obtained by graft copolymerization of styrene to the vinyl group of polydimethylsiloxane-polymethylvinylsiloxane block copolymer or methylvinylsiloxane-dimethylsiloxane copolymer by radical or <sup>50</sup>Co radiation as described in a paper by P. Bajaj, D.C. Gupta and K. Varshung (Polymer Engineering and Science, Vol. 23, p. 825 (1983)) or a paper by J.A. Barrie and K. Munday (Journal of Membrane Science, Vol. 13, p. 175 (1983)). In this method, however, control of the molecular weight of each polystyrene unit and polysiloxane unit and also the molecular weight of the final products are difficult and it is difficult to obtain products having the desired compositions since the method is sometimes accompanied by a crosslinking reaction and it is difficult to maintain proper reaction conditions, furthermore since the graft copolymers have a molecular structure of styrene graft copolymerized to polysiloxane chains there arises a problem in miscibility when the graft copolymers are blended with styrene resins having no other polysiloxane chain in the constituent unit and reduction of impact strength is induced, and the anticipated effect on mold release property in molding of final products, wear resistance, impact resistance, etc. cannot be obtained.

The present inventors had assiduously investigated improvement of polystyrene resin by paying attention to problems in the surface characteristic and impact strength of polystyrene resins and, as a result, reached the present invention.

Namely, the purpose of the present invention is to solve the aforementioned problems and to provide polystyrene resins having improved mold release property in molding, wear resistance, lubricating property, impact resistance, etc.

#### Means to solve the problems

The method of the present invention for preparation of modified polystyrene resins is characterized in that a polystyrene elastomer modified by maleic anhydride (hereinafter abbreviated by modified polystyrene elastomer) is reacted with polysiloxane having a reactive group.

The modified polystyrene elastomer in the method for preparation of modified polystyrene resin of the present invention is obtained by selectively hydrogenating the polybutadiene segment of a styrene-butadiene block copolymer (containing at least one polystyrene segment and one polybutadiene segment) and graft modifying with maleic anhydride.

The first preparation method of impact-resistant polystyrene resin compositions of the present invention is characterized in that modified polystyrene resins of the present invention are mixed with polystyrene or styrene copolymer resins.

The second preparation method of impact-resistant polystyrene resin compositions of the present invention is characterized in that a modified polystyrene elastomer is reacted with polysiloxane having reactive groups in the presence of a polystyrene or styrene copolymer resin.

The polystyrene segment of the styrene-butadiene block copolymer in the present invention is preferably in a range of 15-60 wt% and further preferably 20-50 wt%. If the content of polystyrene segment is less than 15 wt%, there arise such problems that the miscibility of reaction products between modified elastomer and reaction group-containing polysiloxane with polysiloxane or styrene copolymer resins is reduced and the reaction does not sufficiently progress in the second preparation method of compositions of the present invention, on the other hand, if it exceeds 60 wt%, the melting point rises or dispersibility is lowered during the reaction of modified styrene elastomer with reaction group-containing polysiloxane, adversely influencing moldability or final products.

The modified polystyrene elastomer in the present invention is obtained by selectively hydrogenating the polybutadiene segment of a styrene-butadiene block copolymer and graft modifying with maleic anhydride. More specifically, level of unsaturated bonds in the polybutadiene segment without hydrogenating is 20% or less, preferably 10% or less, and further preferably 5% or less. If the unsaturation degree exceeds 20%, heat durability, thermal aging resistance, and weathering resistance are reduced, and it is not preferred. In addition, if the polystyrene segment is hydrogenated by 10% or more, the mechanical strength and impact resistance of compositions of the present invention tend to be lowered.

The content of maleic anhydride in the modified polystyrene elastomer of the present invention is 0.02-20 wt%, preferably 0.1-10 wt%, and further preferably 0.2-5 wt%. If the content of maleic anhydride units is less than 0.02 wt%, it becomes difficult to react the maleic anhydride units of the modified polystyrene elastomer with reactive polysiloxane. In addition, if it exceeds

20 wt%, it is feared that unreacted acid anhydride groups will cut off the modification by polysiloxane chains and reduce the sliding characteristics such as lubricating property of the molding surface and adversely affect the mold release property, wear resistance, and lubricating property during the molding process.

Concrete examples of styrene-butadiene block copolymers used in the present invention are described in U.S. Patent Nos. 4,219,627, 3,251,905, 3,390,207, and Japanese Kokoku Patent No. Sho 45[1970]-29669, etc. In those patents, block copolymers comprising one polystyrene segment and one polybutadiene segment each, which are prepared by successive addition of monomer, addition augmentation of monomer, or coupling reaction, are disclosed.

In addition, they are also described in Japanese Kokoku Patent Nos. Sho 58[1983]-10413, Sho 53[1978]-15958, No. Sho 41[1966]-8782, Japanese Kokai Patent Application No. Sho 53[1978]-94584, U.S. Patent Nos. 3,251,905 and 3,639,521. In these specifications, multiblock copolymers containing tapered copolymer blocks, which are prepared by copolymerizing a mixture of butadiene and styrene utilizing the difference in the copolymerization reaction rate, are disclosed.

The hydrogenation method of styrene-butadiene block copolymers is described in U.S. Patent No. 3,113,986 and Japanese Kokoku Patent No. Sho 59[1984]-37294. Namely, styrene-butadiene block copolymer and multiblock copolymer obtained by the aforementioned method are dissolved in an inert hydrogen diluent, e.g., cyclohexane, and hydrogenation is carried out in the presence of a soluble hydrogenation catalyst.

The known method for graft modification with maleic anhydride is described, for example, in Japanese Kokai Patent Application Nos. Sho 61[1986]-76518, Sho 62[1987]-250018, Sho 62[1987]-79211, etc. For example, maleic anhydride and the aforementioned hydrogenated block copolymer or multiblock copolymer are melt-kneaded in the presence of a radical initiating agent to graft maleic anhydride onto the copolymer.

The reactive polysiloxane used in the present invention has amino group-containing substituents at one end or both ends and a weight-average molecular weight of 1000-100,000 and preferably 5000-50,000. If the molecular weight is lower than 1000, the formation of a three-dimensional member by reaction with modified polystyrene elastomer occurs, the melting point rises, or dispersibility decreases, adversely affecting the moldability of final products. In addition, if the weight-average molecular weight of the reactive polysiloxane is greater than 100,000, it is feared that the reactivity with modified polystyrene elastomer will decrease.

The weight-average molecular weight can be measured by gel permeation chromatography. If the reactive polysiloxane has amino group-containing substituents at one end or both ends, its manufacturing method is not specifically important, but those with a living polymer polysiloxane as the skeleton are especially preferred from the viewpoint of ease of

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control of polymerization degree and molecular weight distribution. The reactive polysiloxane is prepared by living polymerization of hexamethylcyclotrisiloxane in the presence of a lithium catalyst using trimethylsilanol or silanol-terminated dimethylsiloxane oligomer as an initiator, reacting with dimethylchlorosilane to introduce Si-H bonds to one end or both ends, and hydrosilylating to convert one end or both ends into amino groups.

The modified polystyrene resins of the present invention are manufactured by reacting a modified polystyrene elastomer with an amino group-terminated reactive polysiloxane by the solution method or melt method. The addition ratio of the reactive polysiloxane to the modified polystyrene elastomer in the above reaction is preferably 0.05-70 wt%. If it is less than 0.05 wt%, the surface lubricating property and wear resistance of molded products by modified polystyrene resins will be insufficient, and if it exceeds 70 wt%, it is feared that undesirable effects will result as follows: the modified polystyrene resins will become greasy, the miscibility of molded articles from the resins will decrease, bleeding will occur, deterioration of physical property will be induced, etc. When the modified polystyrene resins of the present invention are manufactured by the solution method, the aforementioned modified polystyrene elastomer and reactive polysiloxane having amino substituents at one end or both ends are stirred in a common solvent, and the solvent is removed to obtain the desired products. As solvents used in this case, linear or cyclic ethers such as diisopropyl ether, di-n-propyl ether, dioxane, tetrahydrofuran, etc., aliphatic or aromatic ketones such as acetone, methyl ethyl ketone, methyl isobutyl ketone, acetophenone, cyclohexanone, etc., esters such as ethyl acetate, isobutyl acetate, n-butyl acetate, etc., and aromatic hydrocarbons such as toluene, xylene, etc., are examples. Other solvents beside those illustrated above can be used if they are common solvents for modified polystyrene elastomer and reactive polysiloxanes having amino groups at one end or both ends, and they do not cause any problems in safety and hygiene. In addition, they can be used as mixed solvents of two or more.

In addition, room temperature is sufficient for the reaction temperature, but heating the solvents up to the boiling temperature of the solvents does not cause any problem.

The first manufacturing method of impact-resistant polystyrene resin compositions of the present invention is a method of mixing modified polystyrene resins of the present invention with polystyrene or styrene copolymer resin by the melt method. It is possible to mix them using melt kneaders, which are used in the general synthetic resin field, e.g., an extruder, and in this case the kneading temperature is 250°C or lower, preferably 150-230°C. If the temperature is higher than 250°C, it is feared that the stability of modified polystyrene resins may be damaged. Various additives such as thermal stabilizer, UV absorbers, lubricant, coloring agent, and antistatic agent, or various fillers such as tale, glass fiber, etc., can be added during mixing.

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The second manufacturing method of compositions of the present invention is a method of simultaneously carrying out the manufacture of modified polystyrene resins and mixing with styrene polymers, namely a modified polystyrene elastomer is mixed with a reactive polysiloxane having amino groups at one end or both ends and polystyrene or styrene copolymer resin, melted, and reacted, or these can be dissolved in a common solvent and reacted under heating, and then the solvent is removed. At this time, various additives can be added as in the first manufacturing method.

As the polystyrene used in the manufacturing method of impact-resistant polystyrene compositions, polystyrene and high-impact polystyrene are examples. As styrene copolymer resins, acrylonitrile-styrene copolymer, acrylonitrile-butadiene-styrene copolymer, acrylonitrile-styrene-acrylic rubber copolymer, acrylonitrile-styrene-EPDM (ethylene-propylene-diene terpolymer) copolymer, acrylonitrile-chlorinated polyethylene-styrene copolymer, methyl methacrylate-butadiene styrene copolymer, styrene-butadiene-ethylene-styrene block copolymer, etc., are examples. Basically, those containing styrene as the polymerization unit can be used without any specific restriction, but those containing less amounts of acid anhydride groups such as maleic anhydride group and groups having high polarity and high reactivity, such as a carboxyl group, in the polymerization unit are preferred. In addition, two or more styrene polymers can be mixed and used.

#### Application examples

Hereinaster, the present invention will be explained by application examples.

The physical properties in the application examples were determined by molding the impact-resistant polystyrene resin compositions into test pieces using an injection molding machine with a cylinder temperature of 180-200°C and measuring the physical properties according to the following methods.

Tensile test: ASTM D638 Bending test: ASTM D790

Dynamic friction coefficient: JIS K7218A

(counter material SUS304, velocity 4 cm/s, load 2 kgf, holding time 30 min)

Friction wear: JIS K7218A

(counter material SUS304, velocity 50 cm/s, load 1.02 kgf, holding time 100 min)

Notched izod impact strength: ASTM D790

#### Application Example 1

First, 50 parts by weight of modified polystyrene elastomer, obtained by selectively hydrogenating the butadiene of a 2 wt% maleated styrene-butadiene-styrene copolymer having specific gravity 0.91 g/cm<sup>3</sup>, styrene content 28 wt%, and solution viscosity 6000 cP at 25°C in 20 wt% toluene, were dissolved in 700 parts by weight of tetrahydrofuran, and 50 parts by weight of amino group-terminated (at one end) polydimethylsiloxane having molecular weight (by gel permeation chromatography) of 5000 were added and reacted under stirring for 15 h. Then, tetrahydrofuran was removed under reduced pressure, and the residual solid material was frozen, crushed, and vacuum-dried to obtain a modified polystyrene resin of the present invention having the structural formula shown below.

# Application Examples 2-4

Granular high-impact polystyrene having a melt index (200°C, 5000 g), measured according to ASTM D1238, of 3.5, modified polystyrene elastomer used in Application Example 1, and amino group-terminated (at one end) used in Application Example 1 were mixed at composition ratios shown in Table 1, extruded by an extruder at a cylinder temperature of 200-220°C, and pelletized to obtain impact-resistant polystyrene compositions. The physical properties were measured and results are shown in Table 1.

#### Application Example 5

An impact-resistant polystyrene resin composition was obtained by extruding 6 parts by weight of modified polystyrene resin used in Application Example 1 and 94 parts by weight of high-impact polystyrene resin, the same as that used in Application Examples 2-4, in an extruder at a cylinder temperature of 200-220°C. The physical properties were measured and results are shown in Table 1.

#### Application Examples 6-8

Impact-resistant polystyrene resin compositions were obtained by mixing high-impact polystyrene resin used in Application Examples 2-4, the same modified polystyrene elastomer as that used in Application Examples 2-4, and amino group-terminated (at both ends) reactive polysiloxane having weight-average molecular weight of 10,000 and the structural formula shown below, and extruding in an extruder at a cylinder temperature of 200-220°C. The physical properties are shown in Table 1.

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### Comparative Example 1

The physical properties of the high-impact polystyrene resin used in Application Examples 2-6 are shown in Table 1.

#### Comparative Example 2

A resin composition was obtained by mixing 97 parts by weight of high-impact polystyrene resin used in Application Examples 2-6 with 3 parts by weight of polysiloxane having no reactive group and weight-average molecular weight of 10,000, extruding in an extruder at a cylinder temperature of 200-220°C, and pelletizing. The physical properties were measured and results are shown in Table 1.

# Comparative Example 3

A resin composition was obtained by mixing 94 parts by weight of high-impact polystyrene used in Application Examples 2-6, 3 parts by weight of polysiloxane having no reactive group and weight-average molecular weight of 10,000, and 3 parts by weight of modified polystyrene elastomer used in Application Examples 1-4, extruding in an extruder at a cylinder temperature of 200-220°C, and pelletizing. The physical properties were measured and results are shown in Table 1.

Table 1

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Key: 1 Example

- Application example 2
- Comparative example 3
- Addition amount (wt%) 4
- Amino group-terminated (at one end) 5
  - polydimethylsiloxane
- 6 Amino group-terminated (at both ends) polydimethylsiloxane
- 7 8 Polysiloxane having no reactive group
- Modified polystyrene clastomer
- Modified polystyrene resin of Application Example 1 9
- High-impact polystyrene 10
- Physical properties 11
- 12 Tensile elastic modulus
- 13 Flexural elastic modulus
- Notched izod impact strength 14
- Dynamic friction coefficient 15
- 16 Frictional wear

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#### Effect of the invention

The advantage of the present invention is to obtain modified polystyrene resins with a clearly controlled structure by a method of reacting modified polystyrene elastomer with reactive polysiloxane, and this solves impact strength of conventional polystyrene resins and unsolved problems during molding all at once.

It was made possible to intensify the impact characteristic and improve the mold release property in molding, wear resistance, lubricating property, and sliding characteristic of the molding surface by containing the modified styrene resins of the present invention in conventional polystyrene resins. This is because the modified styrene resins of the present invention are excellent elastomers and have excellent miscibility with respect to polystyrene resins.

Since in the modified styrene resins of the present invention, their structure, especially the structure of the polysiloxane part, can be more easily controlled than in the modification method of the prior art, optimum control of the molecular weight of the polysiloxane part and the ratio of the polystyrene elastomer part to the polysiloxane part is possible according to the purpose, and the modified styrene resins can comply with the diverse in requirements of the wide uses of styrene thermoplastic elastomers.

Since polystyrene resins are inexpensive, hard, have excellent water resistance and high frequency electric insulating property, are resistant to acids and alkali, and can be easily injection molded, their uses are extremely wide and the modified polystyrene compositions of the present invention can comply with the diverse requirements for improvement of conventional polystyrene resins.